

Monsanto

MIC ENVIRONMENTAL SCIENCES
(CO./DIV./DEPT./LOCATION)

SPECIAL STUDY REPORT
(TYPE OF REPORT)

REPORT NO.: ES-80-SS-27

JOB/PROJECT NO.: 43-000-760.26-8623045
Transfer to 02-003-913.41-610

DATE: October 31, 1980

TITLE: MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE
DEAD CREEK SITE - QUALITY ASSURANCE

AUTHORS: R. G. Kaley, II and O. Hicks

ABSTRACT: This report summarizes the quality assurance results obtained for various samples analyzed during the course of this project. The accuracy (recovery from spiked samples) and precision (relative standard deviation of replicate determinations) results are tabulated herein. Although it would be difficult to summarize the overall performance of the methods for all the analytes, in general, the methods performed at the recovery and precision levels established during method validation.

DISTRIBUTION: ~~PAUL Heister~~ 1740

O. Hicks - NIE
R. G. Kaley - NIE
J. P. Mieure - N3A
R. H. Sinise - 1740
C. R. Sisco - NIE

WGK 1111041

COMPANY CONFIDENTIAL

This document is the property of Monsanto Company and the recipient is responsible for its safekeeping and disposition. It contains CONFIDENTIAL INFORMATION which must not be reproduced, revealed to unauthorized persons or sent outside the Company without proper authorization.

EPA/CEPRO COPPER/EIL/PCB ATTORNEY WORK PRODUCT / ATTORNEY CLIENT PRIVILEGE

R. G. Kaley, II and O. Hicks
MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE
DEAD CREEK SITE - QUALITY ASSURANCE

REPT. NO.: ES-80-SS-27
AUTHORS:
TITLE:

COPY NO.:

7-10881R1

7-10881A

MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE - QUALITY ASSURANCE

INTRODUCTION

Following media reports and subsequent Illinois EPA concern about hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant sampled several areas at the site. Samples were submitted to Environmental Sciences for analyses for polychlorinated biphenyls, elemental phosphorus, chlorinated benzenes, chlorinated phenols, phosphate esters, and metals. During the various determinations, replicates and spiked samples were analyzed to evaluate the performance of the method used for these particular samples.

SUMMARY

This report summarizes the quality assurance results obtained for various samples analyzed during the course of this project. The accuracy (recovery from spiked samples) and precision (relative standard deviation of replicate determinations) results are tabulated herein. Although it would be difficult to summarize the overall performance of the methods for all the analytes, in general, the methods performed at the recovery and precision levels established during method validation.

DETAILS

WGK 1111042

Analytical Methods

The soil samples were analyzed for the various chemicals using established procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

Analyte	Method No.	Title
Polychlorinated Biphenyls	ES-80-M-28	Determination of Polychlorinated Biphenyls in Soil and Sediment
Chlorinated Benzenes	ES-80-M-29	Determination of Chlorinated Benzenes in Soil and Sediment
Chlorinated Phenols	ES-80-M-30	Determination of Chlorinated Phenols in Soil and Sediment
Elemental Phosphorus (P ₄)	ES-80-M-24	Determination of Elemental Phosphorus (P ₄) in Soil and Sediment
Phosphate Esters	ES-80-M-5	Determination of Group I Compounds in Sediments . . .
Metals	Ref. 1, 2	Inductively Coupled Plasma (ICP) . . . Method for Trace Element Analysis of Water and Wastes
Arsenic	Ref. 3	Methods for Chemical Analysis of Water and Wastes - Arsenic

All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

Results

The results for the determinations of the compounds of interest have been reported in Special Studies ES-80-SS-24, 25, and 26, Measurement of Selected Chemicals in Soil from the Dead Creek Site . . . This Special Study is a compilation of the quality assurance results for all three Special Studies.

Quality Assurance

The recovery and precision results for the determinations are tabulated in Tables I-V. Each table contains the results for all quality assurance samples for a specific group of compounds. Recovery results are reported as percent recovery, calculated as

$$\% \text{ Recovery} = \frac{\text{Concentration (sample + spike)} - \text{average concentration (sample)}}{\text{Concentration (spike added)}} \times 100$$

Precision results are reported as percent relative standard deviation (RSD) for replicate determinations.

The tables present the recovery and precision results in concentration ranges (1-10 ppm to 10,000 - 100,000 ppm). The entries are averages of all values for all samples which had either recovery or precision evaluated in that range. All values are for actual samples except the metals recovery results, which are for spiked blank soil. In the recovery column, NE means Not Evaluated, i.e., no samples were spiked in that concentration range, and ND means Not Determinable, i.e., the spiking level was too low (usually <50%) compared to the level actually in the sample. In the precision columns, NE means Not Evaluated, i.e., no replicates were analyzed which contained the analyte in that concentration range.

More detailed compilations of the accuracy and precision results can be found in Reference 4.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals - 6, Section 4.1.3.
2. Federal Register, Vol. 44, No. 233, December 3, 1979.
3. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 206 - Arsenic, pages: 206.2-1 to 206.5-2.
4. RGK NBP 1914831

WCK 1111043

RECOVERY AND PRECISION

TABLE I. PCBs AND ELEMENTAL PHOSPHORUS (P₄) IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000 ppm		10,000-100,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
PCB's (Cl ₂ to Cl ₆ Homologs)		ND	NE	70%	17%	120%	18%	77%	58%	NE	0%
P ₄		56%	NE	NE	NE	NE	NE	NE	NE	NE	NE

WCK 1111044

RECOVERY AND PRECISION

TABLE II. CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
MONOCHLOROBENZENE		105%	NE	110%	NE	100%	NE
P-DICHLOROBENZENE		120%	21%	125%	64%	120%	NE
O-DICHLOROBENZENE		125%	16%	120%	NE	120%	8%
TRICHLOROBENZENES (3)		96%	14%	110%	13%	120%	NE
TETRACHLOROBENZENES (3)		110%	9%	120%	NE	130%	NE
PENTACHLOROBENZENE		140%	12%	120%	NE	140%	NE
HEXACHLOROBENZENE		135%	13%	90%	NE	110%	NE
NITROCHLOROBENZENES (O-,P-)		125%	37%	120%	NE	120%	26%

WCK 1111045

RECOVERY AND PRECISION

TABLE III. CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
O-CHLOROPHENOL		19%	34%	64%	NE	58%	NE
P-CHLOROPHENOL		36%	26%	16%	NE	30%	NE
2,4-DICHLOROPHENOL		66%	47%	59%	NE	50%	NE
PENTACHLOROPHENOL		140%	46%	40%	NE	36%	20%

W6K 1111046

RECOVERY AND PRECISION

TABLE IV. PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL		1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
DIBUTYLPHENYL PHOSPHATE	75%	NE	130%	NE	120%	12%	NE	NE	NE	NE
BUTYLDIPHENYL PHOSPHATE	120%	42%	115%	NE	NE	NE	NE	NE	NE	NE
TRIPHENYL PHOSPHATE	120%	89%	120%	NE	115%	NE	NE	NE	NE	6%
2-ETHYLHEXYLDIPHENYL PHOSPHATE	90%	47%	110%	NE	115%	NE	NE	NE	NE	NE
ISODECYLDIPHENYL PHOSPHATE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
T-BUTYLPHENYLDIPHENYL PHOSPHATE	70%	NE	92%	100%	100%	NE	NE	NE	NE	NE
DI-T-BUTYLPHENYLPHENYL PHOSPHATE	88%	NE	96%	NE	NE	NE	NE	NE	NE	NE
NONYLPHENYLDIPHENYL PHOSPHATE	84%	8%	76%	NE	96%	NE	NE	NE	NE	NE
CUMYLPHENYLDIPHENYL PHOSPHATE	62%	21%	76%	NE	88%	NE	NE	NE	NE	NE

MCK 1111047

RECOVERY AND PRECISION

TABLE V. METALS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		1-100 ppm		100-1,000 ppm		1000-10,000 ppm		10,000-100,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
SILVER		NE	NE	NE	46%	NE	NE	NE	NE	NE	NE
ALUMINUM		ND	NE	ND	NE	ND	NE	NE	6.5%	NE	NE
BARIUM		ND	NE	ND	NE	89%	37%	NE	0%	NE	NE
BERYLLIUM		98%	NE	98%	NE	94%	NE	NE	NE	NE	NE
BORON		0%	NE	65%	19%	81%	3%	NE	NE	NE	NE
CALCIUM		ND	NE	ND	NE	ND	NE	NE	8.3%	NE	7.5%
CADMIUM		89%	5.2%	97%	21%	96%	NE	NE	NE	NE	NE
COBALT		51%	NE	115%	6.5%	97%	5.1%	NE	NE	NE	NE
CHROMIUM		27%	NE	109%	20%	91%	6.4%	NE	NE	NE	NE
COPPER		0%	NE	143%	66%	90%	NE	NE	NE	NE	11%
IRON		ND	NE	ND	NE	ND	NE	NE	NE	NE	8.1%
MAGNESIUM		ND	NE	ND	NE	ND	NE	NE	7.8%	NE	NE
MANGANESE		ND	NE	ND	13%	ND	10%	NE	NE	NE	NE
MOLYBDENUM		53%	NE	83%	11%	83%	NE	NE	NE	NE	NE
SODIUM		ND	NE	ND	NE	ND	11%	NE	NE	NE	NE
NICKEL		0%	NE	108%	14%	91%	13%	NE	4.5%	NE	NE
LEAD		0%	NE	165%	21%	93%	NE	NE	6.5%	NE	NE
PHOSPHORUS	MGR 1111048	ND	NE	ND	NE	ND	10%	NE	17%	NE	7.9%
ANTIMONY		0%	NE	27%	2.9%	27%	13%	NE	NE	NE	NE
SILICON		ND	NE	ND	NE	0%	49%	NE	NE	NE	NE
TIN		88%	NE	85%	5.6%	96%	5.4%	NE	NE	NE	NE
STRONTIUM		81%	NE	105%	3.3%	94%	6.5%	NE	NE	NE	NE
TITANIUM		ND	NE	99%	30%	30%	1.3%	NE	NE	NE	NE
VANADIUM		ND	NE	ND	13%	120%	11%	NE	NE	NE	NE
ZINC		ND	NE	139%	34%	87%	NE	NE	8.9%	NE	16%
ARSENIC (By AA)		NE	NE	NE	NE	NE	NE	NE	NE	NE	NE

Submitted by:

Monsanto Industrial Chemicals Company
Environmental Sciences Section - N1E
800 North Lindbergh Boulevard
St. Louis, Missouri 63166

Prepared by:

Robert G. Kaley, IV
Robert G. Kaley, IV
Research Group Leader

Approved by:

James P. Mieure
James P. Mieure
Manager, Environmental Sciences

WGK 1111049